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LUCAS DO AMARAL COLOMBO

Resistência da união de materiais restauradores CAD/CAM tratados com diferentes protocolos de condicionamento de superfície

Bond strength of CAD/CAM restorative materials treated with different surface etching protocols

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Dissertação apresentada à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas como parte dos requisitos exigidos para a obtenção do título de Mestre em Materiais Dentários.

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Orientador: Prof Dr Mario Fernando de Goes

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A Ata da defesa com as respectivas assinaturas dos membros encontra-se no processo de vida acadêmica do aluno.

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RESUMO

O objetivo neste estudo foi avaliar diferentes protocolos de tratamento de superfície de materiais cerâmicos CAD/CAM em relação à morfologia de superfície e resistência da união entre cerâmica e cimento resinoso. Foram elaborados protocolos de tratamento de superfície(n=10) baseados nas recomendações do fabricante, variando a concentração e o tempo de aplicação do ácido fluorídrico seguido pela aplicação de silano(HF5%20s+S, HF5%60s+S, HF10%20s+S, HF10%60s+S), assim como o uso um primer cerâmico autocondicionante(MBEP), além de um grupo controle(CT). Estes tratamentos foram executados em três materiais cerâmicos: cerâmica à base de dissilicato de lítio(IPS e.max CAD), à base de leucita(IPS Empress CAD) e cerâmica infiltrada por polímero(ENAMIC). As cerâmicas foram fixadas com cimento resinoso a um bloco de resina composta para CAD/CAM(Lava Ultimate) e seccionadas em uma cortadeira de precisão, sob irrigação constante, até a obtenção de espécimes em forma de palitos. Todos os espécimes foram submetidos ao ensaio de microtração, sob velocidade de 0,5mm/min. A análise morfológica da superfície cerâmica tratada e o modo de fratura foram avaliados com microscópio eletrônico de varredura. Todos os dados foram submetidos à análise de variância à dois fatores e ao teste de Tukey (α=0.05). Os resultados mostraram que todos os tratamentos aumentaram significativamente a resistência da união em comparação com o grupo controle. Entretanto, as variações da concentração e do tempo de aplicação do ácido fluorídrico não influenciaram significativamente a resistência da união em nenhum dos materiais avaliados, sendo inclusive estatisticamente iguais ao grupo tratado com primer cerâmico autocondicionante. A cerâmica infiltrada por polímero foi o material que apresentou valores médios significativamente maiores de resistência da união, enquanto as ceramicas à base de dissilicato de lítio e à base de leucita não apresentaram diferenças estatisticamente significativas entre si. O padrão de fratura mais prevalente em todos os grupos foi falha mista, seguido por falha coesiva no cimento resinoso. A morfologia da superfície da cerâmica apresentou alterações mais evidentes nos grupos de maior concentração e maior tempo de aplicação do ácido fluorídrico. O grupo tratado com o primer cerâmico autocondicionante mostrou alterações mais suaves nas superfícies cerâmicas. Assim, observou-se que todos os tratamentos contribuíram para melhorar a adesão das superfícies cerâmicas ao cimento resinoso. Enquanto a variação da concentração e do tempo de aplicação do ácido fluorídrico não influenciaram nos valores de resistência da união, o primer cerâmico autocondicionante apresentou menor alteração morfológica e mostrou-se tão eficiente quanto os tratamentos convencionais à base de ácido fluorídrico mais silano.

Palavras chave: Cerâmicas CAD/CAM. Condicionamento com ácido fluorídrico. Primer cerâmico autocondicionante.

ABSTRACT

The objective of this study was to evaluate different protocols for surface treatment of CAD/ CAM ceramic materials on the surface morphology and bond strength between ceramic and resin cement. Surface treatment protocols were elaborate based on the manufacturer's instructions, varying the concentration and application time of hydrofluoric acid followed by application of silane(HF5%20s+S, HF5%60s+S, HF10%20s+S, HF10%60s+S), as well as the use of a self-etching ceramic primer(MBEP) and a control group(CT). The same surface treatments were performed on three ceramic materials: lithium disilicate based ceramics(IPS e.max CAD), leucite based ceramics(IPS Empress CAD), and polymer infiltrated ceramics(ENAMIC). The materials were cemented with a resin cement to a block of nanoceramic resin and sectioned using a precision cutter at a speed of 300 rpm and constant water irrigation to obtain stick shaped specimens, which were subjected to microtensile test. The morphological analysis of the fracture was evaluated with a scanning electron microscope. All data were submitted to two-way analysis of variance and Tukey's test ($\alpha = 0.05$). The results showed that all experimental treatments improved bond strength when compared to the control group, however the variation of the concentration and application time of hydrofluoric acid did not influence the bond strength on any of the materials tested, being statistically equal to the group treated with self-etching primer. The polymer infiltrated ceramic was the material that presented higher values of bond strength, and the lithium disilicate and leucite base ceramics showed no statistically significant differences between each other. The most prevalent failure mode in all groups was mixed failure followed by cohesive on the resin cement. The surface morphology analysis was evaluated with a scanning electron microscope. The most evident surface changes were observed in the groups in which the concentration and application time of hydrofluoric acid were higher. The less evident change was noticed on the groups treated with self-etching primer. In conclusion, it was observed that all of the surface treatments tested improved the adhesion of the ceramics to the resin cement, but no differences were observed in this improvement with the variation of the concentration and the time of application of the hydrofluoric acid. In addition, the self-etching ceramic primer proved to be as efficient as conventional hydrofluoric acid plus silane treatments.

Key-words: CAD/CAM ceramics. Hydrofluoric acid etching. Self-etch ceramic primer.

SUMÁRIO

1. INTRODUÇÃO

Cerâmicas odontológicas são materiais que possuem excelentes propriedades mecânicas, ópticas e biológicas (Vanoorbeek *et al*, 2010). Estas características possibilitaram à cerâmica um desempenho superior em relação aos outros tipos de materiais restauradores na maioria de suas indicações e despertou interesse de parte da população. Com o desenvolvimento de novas tecnologias para o processamento destes materiais, como o sistema CAD/CAM (Computer Aided Design/ Computer Aided Manufacturing), este processo tornou-se rápido e conveniente (Kollmuss *et al*, 2016), o que tem tornado estes materiais cada vez mais desejados.

O desenvolvimento dos materiais cerâmicos ao longo dos anos levou a uma melhora nas propriedades. Esta qualificação associada a evolução dos sistemas adesivos possibilitaram a utilização de técnicas onde a cerâmica é aderida ao tecido dental com o uso de agentes resinosos. Esta adesão proporciona uma melhora do desempenho mecânico deste conjunto (Burke *et al*, 2002), o que tem permitido a utilização de peças protéticas de espessura reduzida, asociada às técnicas que envolvem desgaste mínimo da estrutura dental. Contudo, a união dos materiais cerâmicos utilizando adesivos resinosos é um processo complexo que exige tanto o tratamento dos tecidos dentais, como da superfície cerâmica.

Dentro da sua diversidade na composição química (Tian *et al*., 2014), as cerâmicas odontológicas podem ser classificadas de várias formas, como: cerâmicas policristalinas, que possuem baixo ou nenhum conteúdo de matriz vítrea e não são sensíveis ao condicionamento com ácido fluorídrico (HF) em baixas concentrações e nem passíveis de silanização; e cerâmicas vítreas, que possuem alto conteúdo de matriz vítrea e, são sensíveis ao condicionamento com ácido fluorídrico e passíveis de silanização. Dentre os tipos de cerâmicas vítreas estão as reforçadas por dissilicato de lítio, reforçadas por leucita, e cerâmicas híbridas, sendo as últimas compostas de uma fase cerâmica e uma fase resinosa.

O condicionamento com ácido fluorídrico, mesmo em baixas concentrações, é capaz de dissolver a fase vítrea da cerâmica por meio da interação dos íons de fluoreto presentes no ácido com o silício presente na matriz vítrea, levando a exposição das estruturas cristalinas e favorecendo a união mecânica,

além de promover um aumento na energia de superfície (Brentel *et al*., 2007). Já o processo de silanização estabelece uma união química conduzida por uma molécula bifuncional capaz de formar ligações siloxano entre o grupo silanol presente na sua estrutura química e os grupos OH da cerâmica vítrea, além de polimerizar junto com a matriz orgânica do cimento resinoso por meio do grupo polimerizável metacrilato (Tian *et al*, 2014).

Esta forma de tratamento da superfície cerâmica proporciona adesão química e resistência mecânica suficiente para que procedimentos protéticos estéticos retentivos e não retentivos como próteses fixas simples ou múltiplas, além de facetas e laminados cerâmicos possam ser indicados e unidos às estruturas dentais. Este fato aliado às suas excelentes propriedades ópticas determinaram que as cerâmicas vítreas sejam as cerâmicas de eleição para procedimentos estéticos minimamente invasivos (Dirxen C; 2013).

Tem se estabelecido nos últimos anos a tendência de que a combinação das uniões micromecânica e química é considerada ideal para promover longevidade à adesão (Gracis *et al*, 2015). Uma grande vantagem nesta associação está na facilidade técnica de aplicação dos produtos envolvidos, o que viabiliza a sua utilização pelo clínico. Entretanto, diferentes materiais possuem diferentes especificações nos protocolos de condicionamento recomendados pelo fabricante, incluindo variações nas concentrações de ácido fluorídrico, tempo de aplicação e tipos de produtos. Isto pode criar confusão na rotina da prática clínica, além de dúvidas em relação às diferenças na eficácia da adesão entre cada protocolo proposto para cada material.

Diante disto, nos últimos anos tem havido uma busca pela simplificação dos produtos odontológicos com o objetivo de facilitar os procedimentos clínicos. Assim, um primer cerâmico auto-condicionante de um passo foi introduzido no mercado odontológico em 2015. Este primer possui em sua composição um condicionador se superfície (trifluoreto de di-hidrogeno de tetrabutilamônio) menos agressivo que o ácido fluorídrico, combinado com um agente silano, que segundo o fabricante, promete criar condições adequadas para uma união micromecânica e química, proporcionando estabilidade e longevidade da união (Volkel T e Braziulis E, 2015). Contudo, os estudos envolvendo este material ainda são escassos.

Diante do exposto, o objetivo neste estudo foi avaliar o efeito de diferentes protocolos de condicionamento de superfície em três tipos de materiais à

base de cerâmica vítrea, comparando variações de concentração e tempo de aplicação do ácido fluorídrico com , a aplicação do primer cerâmico auto-condicionante, em relação a morfologia de superfície e resistência da união entre as cerâmicas vítreas e o cimento resinoso. As hipóteses testada foram: 1- os distintos protocolos de condicionamento induziriam diferenças na adesão dos cimentos resinosos com as cerâmicas vítreas; 2- o tipo de material testado induziria diferenças na resistência da união e na morfologia de superfície.

Este trabalho foi apresentado no formato alternativo de tese de acordo com as normas estabelecidas pela deliberação 002/06 da Comissão Central de Pós-Graduação da Universidade Estadual de Campinas.

2. ARTIGO

"Bond strength of CAD/CAM restorative materials treated with different surface etching protocols"

ABSTRACT

Clinical Relevance: Different hydrofluoric acid etching concentrations and times of application did not influence the bonding effectiveness. Simplified self-etching ceramic primer produced smoother surface alterations and comparable bonding enhancing effectiveness than separate hydrofluoric acid and silane primer.

Objectives: To Evaluate the effect of different surface treatments on the surface morphology of CAD/CAM ceramics and on the ceramic/cement bonding.

Materials and Methods: 180 cubic sections(4 x 4 x4mm) obtained from three materials (lithium disilicate glass-ceramic- DL (IPS e.max CAD®), leucite-based glass-ceramic- LC (IPS empress CAD®), polymer infiltrated ceramic- EN (VITA Enamic CAD®)) were treated according to the following surface treatments (n=10): 1.No treatment (C); 2.Hydrofluoric acid (5%) applied for 20 seconds plus silane (HF5%20s); 3.Hydrofluoric acid (5%) applied for 60 seconds plus silane (HF5%60s); 4.Hydrofluoric acid (10%) applied for 20 seconds plus silane (HF10%20s); 5.Hydrofluoric acid (10%) applied for 60 seconds plus silane (HF10%60s); 6.Self-etching ceramic primer (MBEP); bonded to resin-ceramic (Lava Ultimate®) cubic sections of same dimensions using resin cement (Variolink N®), stored for 24 hours (n=10), and submitted to microtensile bond strength test (μTBS). Data were analyzed using Two-way ANOVA and Tukey test (α=0.05). Failure pattern and surface morphology were assessed using scanning electron microscopy.

Results: Only individual factors resulted statistically significant for both variables. Regarding μTBS, EN (22.8±8.9 MPa) presented statiscally higher μTBS values than LC (19.0±4.2 MPa) and DL(19.8±6.5 MPa), while treatments HF5%20s (19.8±6.0), HF5%60s(21.2±6.9), HF10%20s(22.1±7.6), HF10%60s(20.8±6.8) and MBEP(20.5±9.7MPa), did not present statiscally different μTBS values among them. But they were statiscally higher than control groups (9.0±3.9 MPa). Interface debonding was detected on C groups for all materials, though EN exhibited better interface performance. MBEP produced smoother surfaces than all HF treatments.

Key-words: CAD/CAM materials, Hydrofluoric acid etching, Self-etching ceramic primer.

INTRODUCTION

Ceramics are widely used materials in dentistry due to their optimal esthetics and mechanical properties [1; 2]. CAD/CAM systems facilitated the manufacturing process of ceramic restorations, making it quick and convenient, as well as minimizing the human factor [3]. This technology encouraged the development of some new restoration materials, widening clinicians' options when performing indirect restorations [4]. With the introduction of improved ceramic materials and adhesion techniques, minimally invasive approach has gained popularity among clinicians and patients, allowing a decrease in dental preparations' extension and as a consequence a reduction in restorations' thickness [5]. Besides improving minimally invasive dentistry, adhesive and luting materials, which may bond ceramic restorations to tooth structures, have also helped to increase ceramics' mechanical behavior [6; 7]. Adhering ceramics to tooth structure and resin-based luting materials is a complex process which requires conditioning of all surfaces involved, specifically on the ceramic side, the most accepted surface treatments are sandblasting, hydrofluoric acid etching and silanization [8]. Although, differences in materials' composition make adhesion protocols widely different and non-specific [9].

Ceramics with high crystalline content (polycrystalline ceramics) require mostly micromechanical treatments, like sandblasting, to promote irregularities in order to increase surface roughness, required for the penetration of resin-based adhesive and luting cements, as well as chemical treatments with MDP based ceramic primers or tribochemical silica coating [10]. Meanwhile, glass-ceramics which show a high glass content (mainly silicon) are prone to hydrofluoric acid (HF) etching as it can chemically remove part of their glassy structure, producing surface irregularities which may serve as micro retentions to luting materials [11; 12].

Moreover, glass-ceramics can be treated using a silane primer. Silane used in dentistry is a bi-functional molecule (siloxane group in one side, which needs to be hydrolyzed, and a methacrylate group in the other side) which can forms a covalent union with ceramics' glassy-phase while polymerizing together with resin-based materials, creating a chemical union between them.[13; 14].

A combined mechanical and chemical surface treatment is considered the gold standard for glass-ceramics as it has shown the most effective and durable results in terms of glass-ceramic/resin-cement bond strength [9; 15]. Alongside with the

abovementioned benefits, HF etching and silanization are quick and simple procedures that can be executed with no need of additional equipment. However, a broad range of surface treatment protocols have been suggested, varying acid concentration and application time in function of each glass-ceramic composition, which can lead to confusion when performing this procedure in the clinical daily practice, as no consensus has been yet achieved in literature regarding the definitive and most adequate HF concentration and application time for each type of glass-ceramic [16].

Within the same direction and to simplify this process, a novel one-step self-etching ceramic primer was developed [17]. This primer mixes on its composition a silane coupler with a mild etchant (tetrabutylammonium dihydrogen trifluoride), which (according to manufacturer) is expected to produce a smoother etching than HF, which in conjunction with the silane agent may provide a strong and long-lasting mechanical/chemical bonding [17]. However, the literature regarding this new approach and its effectiveness is still quite scant.

Thus, the purpose of this study was to evaluate the effect of different etching protocols on the surface morphology and bond strength between resin cement and three vitreous-ceramic based CAD/CAM materials: a lithium disilicate ceramic, a leucite ceramic, and a polymer-infiltrated ceramic (PIC). The null hypotheses tested are that: 1) there are no significant differences on surface morphology and bond strength produced by the surface treatments tested; 2) there are no significant differences on surface morphology and bond strength reported by the different materials tested.

MATERIAL AND METHODS

Preparation of specimens

Three parallelepiped shaped blocks with original dimensions of each material (Table 1) were sectioned transversely and longitudinally to obtain sixty cubic shaped sections with dimensions of 4 x 4 x 4mm, using a diamond impregnated saw (15LC Diamond Series - Isomet Buehler-Microstructural Analysis Division, LakeBluff, Illinois, 60044-USA) positioned in a precision cutter (Isomet 100 - Buehler Ltd., Lake bluuff, IL, USA) at a speed of 300 rpm and constant water irrigation, resulting in a total of 180 sections.

The sections were manually polished using 1000 grit silicon carbide sand paper, also under water irrigation in order to produce a standardized surface on all specimens. All the sections obtained were cleaned under running water and ultrasonicated with deionized water for 10 minutes. Then, all sections were stored in a clean environment and free of moisture.

Experimental groups

The sections of the blocks of each material () were randomly distributed into six groups containing 10 sections each.

Group 1 (Control): No treatment was applied. The specimens were only air-dried for 10 seconds.

Group 2: the specimens were etched with 5% hydrofluoric acid (CEtching - BM4 - Maringá, Brazil) applied to the surface of the material for 20 seconds and washed under running water for 1 minute. The samples were then dried for 10 seconds with oil-free air and then a silane primer (MonoBond N - Ivoclair Vivadent - Schaan, Liechtenstein) was applied, left on for 60 seconds and then blown with oil-free air for 10 seconds.

Group 3: the specimens were etched with 5% hydrofluoric acid (CEtching - BM4 - Maringá, Brazil) applied to the surface of the material for 60 seconds and washed under running water for 1 minute. The samples were then dried for 10 seconds with oil-free air and then a silane primer (MonoBond N - Ivoclair Vivadent - Schaan, Liechtenstein) was applied, left on for 60 seconds and then blown with oil-free air for 10 seconds.

Group 4: the specimens were etched with 10% hydrofluoric acid (Dentsply - York, USA) applied to the surface of the material for 20 seconds and washed under running water for 1 minute. The samples were then dried for 10 seconds with oil-free air and then a silane primer (MonoBond N - Ivoclair Vivadent - Schaan, Liechtenstein) was applied, left on for 60 seconds and then blown with oil-free air for 10 seconds.

Group 5: the specimens were etched with 10% hydrofluoric acid (Dentsply - York, USA) applied to the surface of the material for 60 seconds and washed under running water for 1 minute. The samples were then dried for 10 seconds with oil-free air and then a silane primer (MonoBond N - Ivoclair Vivadent - Schaan,

Liechtenstein) was applied, left on for 60 seconds and then blown with oil-free air for 10 seconds.

Group 6: the specimens were treated with a self-etching ceramic primer (MonoBond Etch&Prime - Ivoclair Vivadent - Schaan, Liechtenstein) which was actively applied for 20 seconds, left on for 40 seconds and washed in running water for 60 seconds (according to manufacturer's instructions).

In order to evaluate surface morphology, other six sections with dimensions of 4 x 2 x 2 mm were obtained from each material, and the respective surface treatment for each group was performed.

Cementation procedure

All ceramic specimens were prepared to be cemented to a pre-polymerized resin composite material. The resin cement (Variolink N - Ivoclair Vivadent - Schaan, Liechtenstein) was manipulated, mixing the base and catalyst pastes following the manufacturer's instructions and dispensed on the ceramic section's surfaces. Subsequently, an equally sized section of the pre-polymerized resin composite (Lava Ultimate CAD - 3M ESPE - Seefeld, Germany), properly treated with following the manufacturer's instructions (Sandblast with aluminum oxide ⩽50µm for 10 seconds, clean under running water, ultrasonicated for 10 minutes, application of adhesive system (ExciTE F DSC - Ivoclair Vivadent - Schaan, Liechtenstein) and light polymerization for 10 seconds using a LED curing light unit (700mW/cm2, BluePhase, Ivoclair Vivadent - Schaan, Liechtenstein) was placed on top of the cement coated ceramic specimen and manually pressed. Then the whole set-up was placed in a mechanical device, where a load of 200g was applied for 10 seconds. Cement excess was removed, and then light polymerized using a LED curing light unit (700mW/cm2, BluePhase, Ivoclair Vivadent - Schaan, Liechtenstein), positioned adjacently to the bonding interfaces, for 20 seconds. The same procedure was repeated on the four sides of the cemented set.

After the cementation procedure, all sets were stored in deionized water at 37 ° C for 24 hours.

Table 1. Materials used in this study

Materials	Composition	Lot nº	Manufacturer
Enamic (EN)	Polymer infiltrated ceramix network UDMA + TEGDMA 14% SiO_2, 58.0 - 63.0% Al_2O_3 20.0 - 23.0% Na_2O 6.0 - 11.0% K_2O 4.0 - 6.0% B_2O_3 0.5 - 2% CaO < 1% TiO_2 < 1% coloring oxides	43230	Vita Zahnfabrik (Bad Säckingen, Germany)
Lava Ultimate	Nano-particulate pre-polymerized resin composite Bis-GMA + UDMA + Bis-EMA + TEGDMA 20% Silica (20 nm), Zirconia (4-11 nm), Zirconia-Silica clusters (0.6-10 µm) 80%	N538336 N354177 N38843 N463323	3M ESPE (Seefeld, Germany)
IPS Empress CAD (LC)	Leucite-based glass-ceramic SiO_2 60.0 - 65.0% Al_2O_3 16.0 - 20.0% K_2O 10.0 - 14.0% Na_2O 3.5 - 6.5% Other oxides 0.5 - 7.0% Pigments 0.2 - 1.0%	U01096	Ivoclair Vivadent (Schaan, Liechtenstein)
IPS e.max CAD (DL)	Lithium disilicate glass-ceramic SiO_2 57.0 - 80.0% Li_2O 11.0 - 19.0% K_2O 0.0 - 13.0% P_2O_5 0.0 - 11.0% ZrO_2 0.0 - 8.0% ZnO 0.0 - 8.0% Al_2O_3 0.0 - 5.0% MgO 0.0 - 5.0% Colouring oxides 0.0 - 8.0%	T25503	Ivoclair Vivadent (Schaan, Liechtenstein)
Variolink N Esthetic Cementation System	Dual-cure resin cement Ytterbiumtrifluoride 25.0% Dimethacrylate 26.3% (Base); 27.9% (Catalyst) Barium glass filler, mixed oxide 48.4% (Base); 46.2% (Catalyst) Initiators and stabilizers 0.3% (Base); 0.8% (Catalyst) Pigments <0.1%	Base: U16084 Catalyst: U48611	Ivoclair Vivadent (Schaan, Liechtenstein)
MonoBond N	Ceramic primer Alcohol solution of silane methacrylate,	U43922	Ivoclair Vivadent

	phosphoric acid methacrylate and sulphide methacrylate.		(Schaan, Liechtenstein)
Monobond Etch and Prime (MBEP)	Self-etching ceramic primer Ammonium polyfluoride + trimethoxypropyl methacrylate - 25.0% Alcohol + Water 75.0 - 85.0%	V09353	Ivoclair Vivadent (Schaan, Liechtenstein)
Hydroflouric Acid Etch Porcelain 10%	Hydrofluoric Acid (HF) 10%	107217H	Dentsply (York, USA)
Power CEtching	Hydrofluoric Acid (HF) 5%	0005/0315	BM4 (Maringá, Brazil)

Bis- GMA, bisphenol A-diglycidyl ether dimethacrilate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; Bis-HEMA, bisphenol A polyethylene glycol diether dimethacrylate. Composition of the materials according to materials safety data sheets (MSDS) provided by the manufacturers.

Microtensile Bond Strength (μTBS) Test

Stick-shaped specimens measuring approximately 1 ± 0.3 mm2, were obtained from each ceramic/resin assembly. The outer sticks from each set as well as those which seemingly presented interface defects were discarded. Therefore, an average of 3 to 5 sticks from each assembly was obtained. The sticks were stored in deionized water at 37° C for 24 hours.

The microtensile test was conducted 48 hours after the cementation procedure. Specimens were individually placed and fixed with a cyanoacrylate gel glue (Super Bonder Gel - Locite Brasil LTDA - São Paulo, Brazil) in a custom jig which was coupled to a universal test machine (EZ-S - Shimadzu - Kyoto, Japan). The specimens were stressed under tensile force until failure at a crosshead speed of 0.5 mm/min. The μTBS was expressed in MPa, derived by dividing the recorded load at failure (N) by the bonded area (mm2). If specimens failed before the actual testing, they were recorded as pre-testing failure.

Statistical Analysis

Data from μTBS were statistically analyzed (Minitab v18 - Minitab Inc. - State College, USA) using two-way ANOVA (surface treatment vs. material) and Tukey post-hoc tests. Pre-testing bond failures were not considered on the statistical analysis.

Failure mode and surface morphology evaluation

The mode of failure for each specimen was determined using light microscope at a magnification of 63X (M35 - Zeiss - Oberkochen, Germany) and classified according to the following categories: Type 1- adhesive failure; Type 2- cohesive failure in the resin cement; Type 3 - mixed failure (cohesive failure in the luting agent and adhesive); Type 4- cohesive failure in ceramic. Then, a representative specimen of each failure mode from each group, was selected, placed on an aluminum stub, sputter-coated with gold-palladium alloy (SCD 050 - Balzers - Schaan, Liechtenstein) and analyzed using a scanning electron microscope (JSM 5600 LV - JEOL - Tokyo, Japan) operating at 15 kV. Each debonded surface was analyzed at various magnifications, and a representative micrograph was taken at magnifications between 65x and 100x in order to illustrate the failure pattern.

For the surface morphology analysis, the sections were mounted on aluminum stubs and sputter coated with gold-palladium alloy (SCD 050 - Balzers - Schaan, Liechtenstein) to be examined using a scanning electron microscope (JSM 5600 LV - JEOL - Tokyo, Japan), operating at: 15 kV and working distance of 20 mm. Representative images of each groups´ typical surface morphology were captured at 2000x magnification.

RESULTS

Tensile bond strength (TBS), failure pattern and interface morphology

Normality (Anderson-Darling test: p=0.316) and homoscedasticity (Bartlett test: p=0.060) were confirmed. Statistical analysis showed that all factors significantly influenced μTBS values (material: p=0.0092; surface treatment: p=0.0014;), while no interaction between factors was found (p=0.2843). μTBS data are displayed on Table 2. Control groups correspondent to DL and LC showed pre-testing failure (100%), while pre-testing failures of 60% were reported for EN. Within the factor “material”, EN obtained the highest μTBS values while DL and LC did not differ among themselves. Regarding surface treatment, all HF treatments and MBEP produced higher μTBS values than control group.

Table 2. Mean microtensile bond strength values (MPa) and standard deviations (parenthesis) from all experimental groups

Material	C	HF5%20S	HF5%60S	HF10%20S	HF10%60S	MBEP	Tukey ($p<0.05$)
DL	-	19.8 (5.3)	16.7 (4.7)	23.7 (8.0)	19.7 (4.4)	19.9 (9.5)	19.8 (6.5) b
LC	-	18.0 (4.0)	20.6 (3.6)	16.8 (3.8)	20.7 (5.2)	18.7 (3.9)	19.0 (4.2) b
EN	9.0 (3.9)	21.6 (7.9)	25.7 (8.6)	26.1 (7.7)	21.6 (9.6)	24.5 (7.8)	22.8 (8.9) a
Tukey ($p<0.05$)	9.0 (3.9) B	19.8 (6.0) A	21.2 (6.9) A	22.1 (7.6) A	20.8 (6.8) A	21.3 (7.3) A	

DL (IPS e.max CAD); LC (IPS Empress CAD); EN (Enamic). Mean values represented with same superscript uppercase letters (row) or lowercase letters (column) are not significantly different according to Tukey's test ($p > 0.05$).

Failure mode prevalence is represented on Figure 1. The most prevalent failure mode was Mixed, followed by cohesive on resin cement. The main failure modes were illustrated on Figures 5; 6 and 7.

Surface morphology analysis (Figures 2; 3 and 4) showed distinguished surface patterns between groups in all materials. Groups treated with MBEP presented mild changes, resulting in surface morphology similar to the Control group. Groups treated with HF in lower concentrations presented significant but fewer surface alterations when compared to higher concentrations, in which a complete surface decharacterization was noticed.

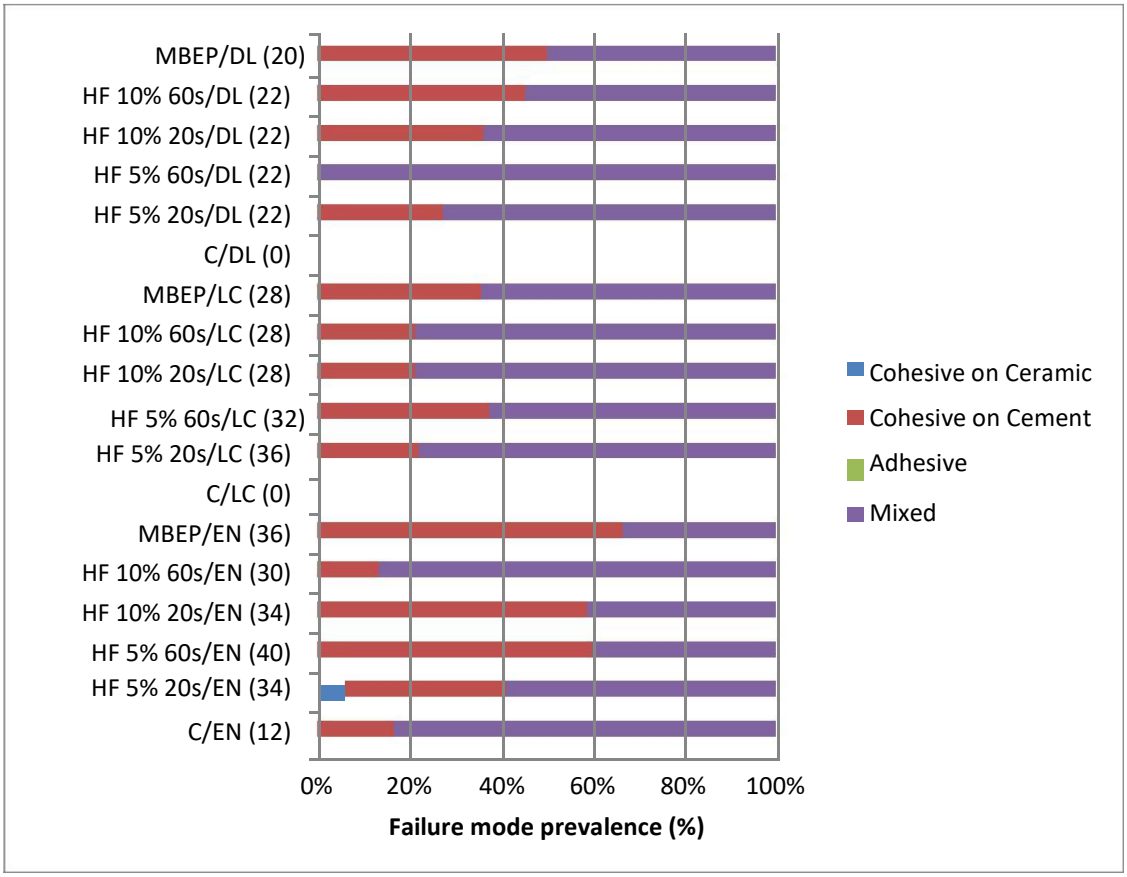

Fig. 1- Results of the SEM failure analysis for all experimental sub-groups. Abreviations: Hydrofluoric acid etching (HF), MonoBond Etch&Prime (MBEP), IPS e-max CAD® (DL), IPS empress CAD® (LC), VITA Enamic (EN), Control Group (C).

DISCUSSION

This in-vitro study evaluated the effect of different glass-ceramic surface treatments on surface morphology and ceramic-cement bonding effectiveness, when varying HF concentration, application time (along with silane) as well as after employing a novel self-etching ceramic primer. In order to do that, different etching protocols were tested based on the most commonly recommended HF concentrations and application times for types of glass-ceramic containing materials, along with a control group. The same etching protocols were tested among all ceramic materials used in this study, independently of manufacturers' instructions. Based on the findings of this study, null hypotheses #1 and 2# must be rejected, since both bond strength and surface morphology were affected by surface treatments and/or materials' type, respectively.

To promote effective adhesion between resin cements and glass-ceramic materials, surface conditioning with hydrofluoric acid (HF) is recommended to produce surface roughness and achieve further micromechanical retention between both materials [18]. HF concentrations between 3% and 10% have been previously shown to promote an adequate surface to fulfill this task [19], therefore the concentrations used on the experimental groups of this study were 5% and 10%, as considered the most widely used within clinicians. The application times of HF used in this study were 20s and 60s, based on manufacturer's instructions, applying both of them to all materials. No higher application time than 60s was employed, based on evidence pointing that etching times higher than 60 seconds generally decrease ceramic-cement bonding ability [20]. Some studies showed that HF treatment alone shows better bond strength values when compared to silane alone on lithium-disilicate, while on leucite and feldspathic, silane alone showed better ones [21]. From this, it can be inferred that luting protocols are material dependent. Nonetheless, the same study has shown that the combination of HF plus silane is the best surface treatment aproach for ceramics in general, although variations on HF concentration and application time were not evaluated [21]. Thus, silane was always applied in our experimental groups using separate application of HF.

The many experimental bonding protocols used in this study showed no statistical differences between each other among all materials tested regarding ceramic-cement bond strength (Tukey $p>0.05$). Even though greater HF concentration and application times may increase the surface roughness [22] and wettability [19] of materials' surfaces. Based on this, it can be inferred that probably there is no direct positive correlation between surface roughness and bond strength. A possible explanation to this is that the higher glassy phase dissolution depth promoted by increased etching times, may be associated with poorer penetration of the resin cement due to its high viscosity, being then difficult to produce increased bond strength among these groups [20]. Additionally, the mechanism of HF etching is not based on acid corrosion of ceramic's glassy matrix – (given the fact that HF is a weak acid in terms of pH) but rather on a chemical interaction between ceramics' silicon and HF fluoride ions [23]. This could mean that as HF etching reaction is ion dependent, increased exposure time of glass-ceramic materials to HF would have no additional benefits.

The effects of HF on the mechanical properties of ceramics are still unclear. While some studies show no significant alterations on the flexural strength of etched ceramics [24;25], others have shown a significant reduction of this mechanical property on lithium-disilicate reinforced ceramics, leucite-reinforced ceramics and feldspathic ceramics, even in lower application times [22;26;27]. In order to avoid the risks of compromising the mechanical strength of restorative materials, milder forms of surface etching must be encouraged. Another known issue of HF is its high toxicity, which may be harmful to clinicians and patients when tissues are directly exposed to it, making it possible to produce various hazardous effects [28].

Following this demand for an alternative product, a novel self-etching ceramic primer (MBEP) was developed. MBEP is a single bottle ceramic primer which allows etching and silanization of glass ceramic surface in one step. It contains a trimethoxypropyl methacrylate for silanization and ammonium polyfluoride for the etching step. Etching pattern created by this novel simplified ceramic primer is less pronounced than that produced by HF, claiming at the same time to be as efficient for bonding as the gold standard procedure (HF along with silane). It provides an easier and faster application sequence due to less steps and is less risky due to the avoidance of HF [17]. Although there are few studies on the literature regarding the efficacy of MBEP, the current work shows promising results so far. One study has shown that while MBEP seems to be the optimal treatment for PIC, HF plus silane, still showed better performance on lithium-disilicate and leucite [29], as opposed to another study , which showed similar performance between MBEP and HF plus silane on lithium-disilicate [30]. In our study, a higher number of pre-testing failures and lower μTBS values were expected on the MBEP groups due to its previously-reported smoother etching pattern (Figures 2;3;4), associated with extra tension received at the interface bonding area of ceramic-resin specimens when sawing them into sticks. However, this situation was not observed, since there were no statistical differences in terms of μTBS and pre-test failures when compared to all other surface treatments tested except Control, within all materials. Therefore, our results are in accordance with the data provided by the manufacturer [17], which claims that this product not only provides additional benefits overall when compared to HF, but performs equally well as of bonding enhancer, although further studies should evaluate its long-term efficacy.

Regarding the materials used in this study, EN showed the highest μTBS values followed by DL and LC which did not differ between them (Table 2). These results agree with another study which reported no statistical difference on bond strength between leucite and lithium disilicate [31]. A possible explanation for the higher μTBS reported by EN when compared to DL and LC is that EN inherently has a rougher surface [8;32], which could result in a more favorable bonding especially on the Control groups. According to a previous work, etching the surface of feldspathic ceramics causes irregular dissolution of glassy phase, creating pits, fissures and undercuts, while on EN, the ceramic matrix is selectively removed, exposing the resin network [16], and revealing a “honeycomb”-like structure (also seen in the current work: Figure 4). This structural configuration may offer a very high potential for micromechanical interlocking [33], as well as the interesting possibility of a chemical affinity between its polymeric matrix and the resin cement, given the fact that both structures are composed of organic polymers, therefore should present some degree of chemical interaction; although this is yet to be confirmed. Another possible explanation may lie on materials’ mechanical properties. While DL and LC have higher modulus of elasticity, EN has been reported to withstand high levels of elongation at high stress before failure, due to its integrated polymer network, which may provide more stability, in spite of a lower modulus of elasticity [34]. This could mean that the stresses generated at the bonded interface could be better spread along the material’s body during the tensile test [35].

Similarly to the bond strength results, the failure mode prevalence observed did not differ between the surface treatments tested. The predominant failure mode was mixed, followed by cohesive on cement on all groups, except groups HF10%20S/EN, HF5%60S/EN and MBEP/EN, which presented mostly cohesive on cement failures. The prevalence of cohesive on cement failure mode on some groups of EN, associated with a higher bond-strength of this material in comparison to the others could mean a stronger adhesion of the resin cement to it’s surface, given the properties of this material discussed above. EN was the only material which presented cohesive failure on ceramic (Figure 1).The prevalence of mixed failure mode as well as the shortage of adhesive failures on experimental groups is a positive finding, pointing to the fact that the material-substrate system fails as one, meaning the absence of a weak link. On the control groups for DL and

LC all specimens presented interface debonding before the μTBS test, which could mean the occurrence of adhesive failures in these groups. Surprisingly, the most prevalent failure mode for the Control group of EN was not adhesive, which lay emphasis on the inherent properties of this material mentioned above.

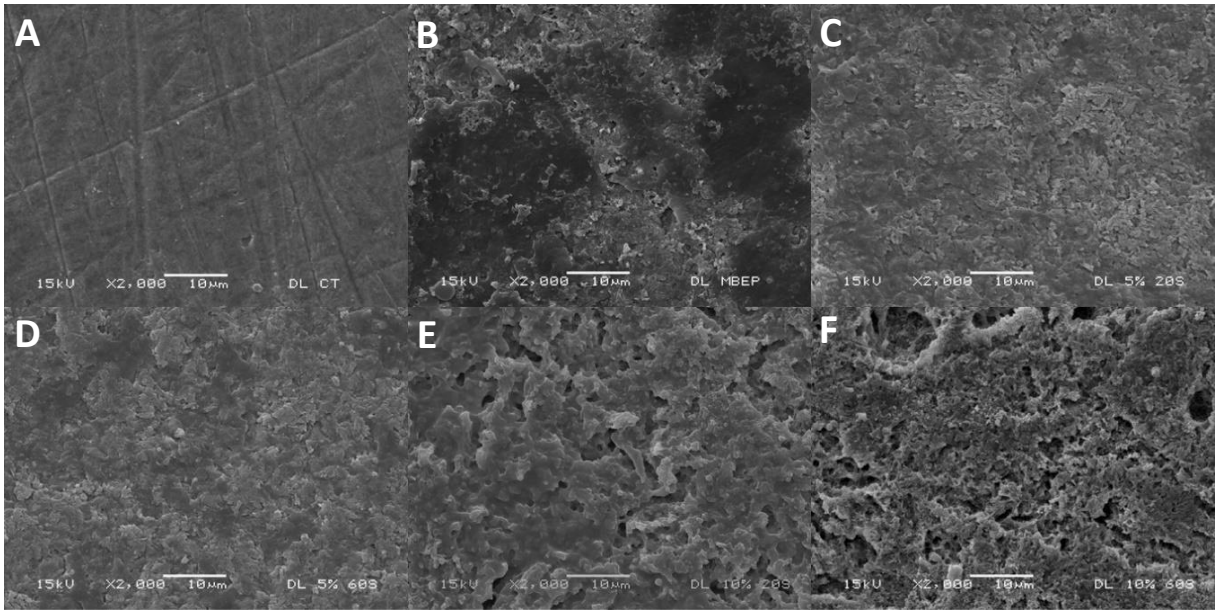

Fig. 2- Images resulting from all surface treatments tested on DL (IPS-e.max CAD). For the treatments tested, from less aggressive to most aggressive (A: Control; B: MBEP; C: HF5%20s; D: HF 5%60s; E: HF 10%20s; and F: HF 10%60s), different etching patterns were found with distinct degrees of vitreous phase dissolution and exposure of lithium disilicate crystals. Image A shows no dissolution of the vitreous phase, image B shows mild dissolution, while there is an increase in the degree of vitreous phase dissolution with higher HF concentrations and application times shown in images C, D E and F.

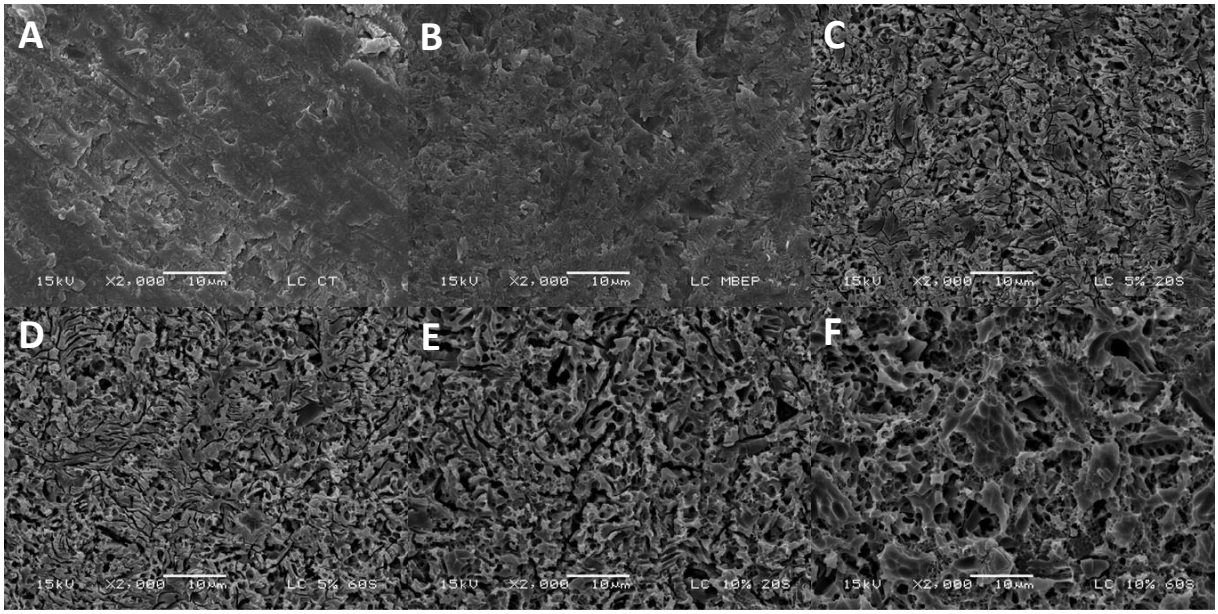

Fig. 3- Images resulting from all surface treatments tested on LC (IPS-empress CAD). For the treatments tested, from less aggressive to most aggressive (A: Control; B: MBEP; C: HF5%20s; D: HF 5%60s; E: HF 10%20s; and F: HF 10%60s), a circular etching pattern was found with distinct degrees of vitreous phase dissolution. Image A shows no dissolution of the vitreous phase, image B shows mild dissolution, while there is an increase in the degree of vitreous phase dissolution with higher HF concentrations and application times shown in images C, D E and F.

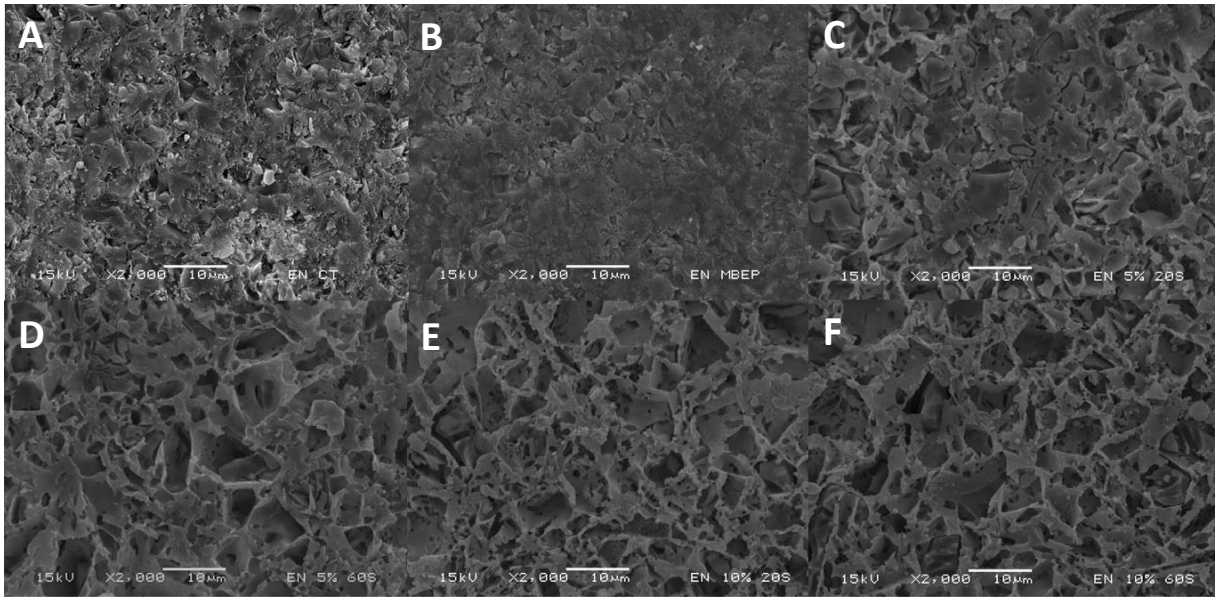

Fig. 4- Images resulting from all surface treatments tested on EN (VITA Enamic). For the treatments tested, from less aggressive to most aggressive (A: Control; B: MBEP; C: HF5%20s; D: HF 5%60s; E: HF 10%20s; and F: HF 10%60s), a selective etching pattern was found with distinct degrees of vitreous phase dissolution and exposure of the “honeycomb-like” polymeric matrix. Image A shows no dissolution of the vitreous phase, image B shows mild dissolution, while there is an increase in the degree of vitreous phase dissolution with higher HF concentrations and application times shown in images C, D E and F.

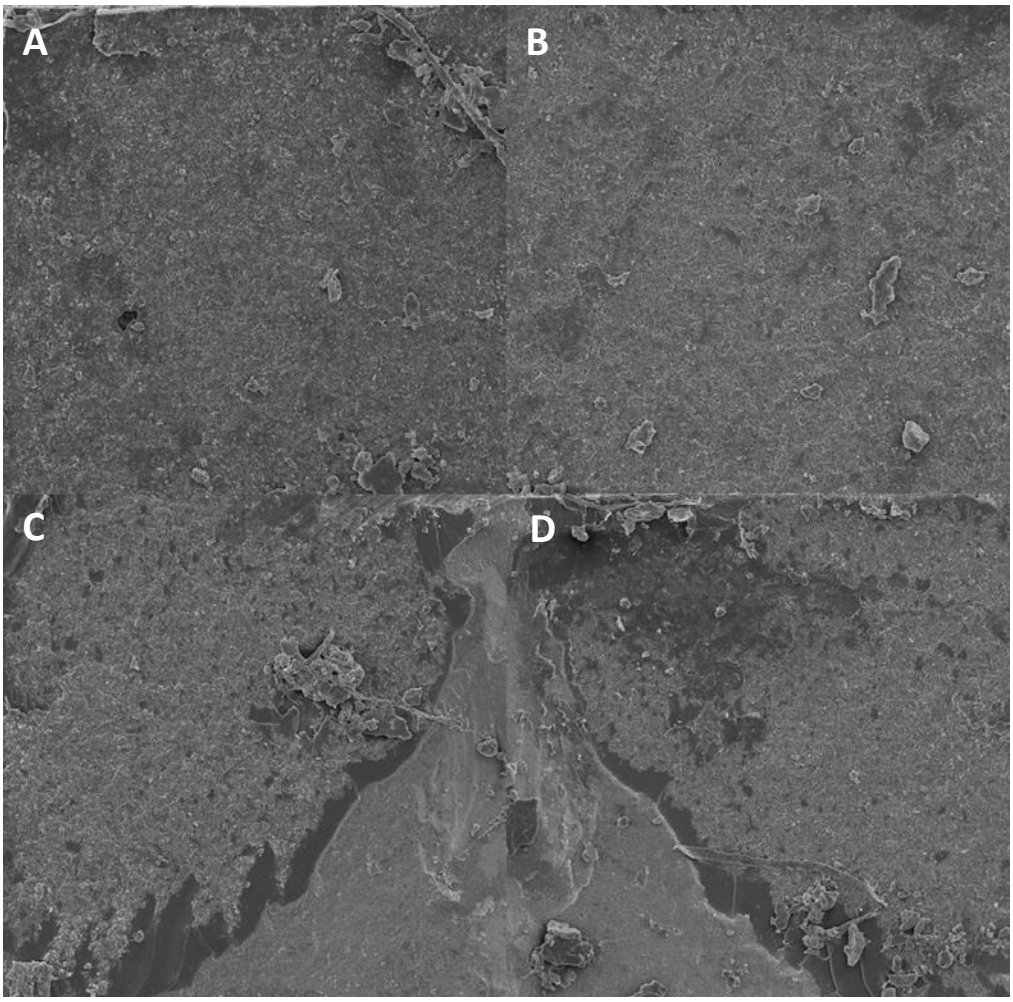

Fig. 5- Fracture pattern on both sides from DL group. Paired images (AB and CD) show the two debonded surfaces of the same stick. No distinction on the fracture patterns between all experimental groups was found. Images A and B show a mainly uniform cohesive failure in resin cement; Images C and D show a mixed fracture pattern, with mainly fractured resin cement sites and some areas of adhesive debonding.

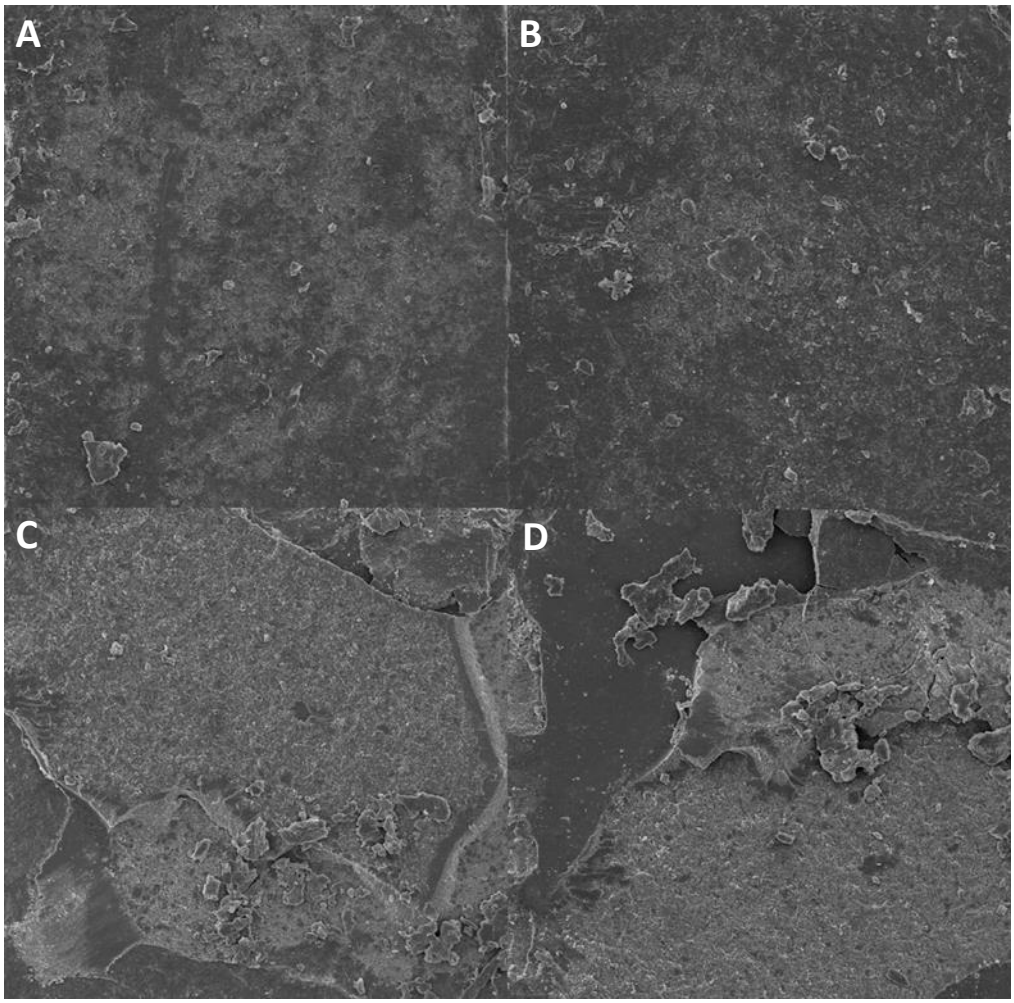

Fig. 6- Fracture pattern on both sides from LC group. Paired images (AB and CD) show the two debonded surfaces of the same stick. No distinction on the fracture patterns between all experimental groups was found. Images A and B show a mainly uniform cohesive failure in resin cement with some resin cement filling particles through the whole fracture area; Images C and D show a mixed fracture pattern, with mainly fractured resin cement sites and some areas of adhesive debonding.

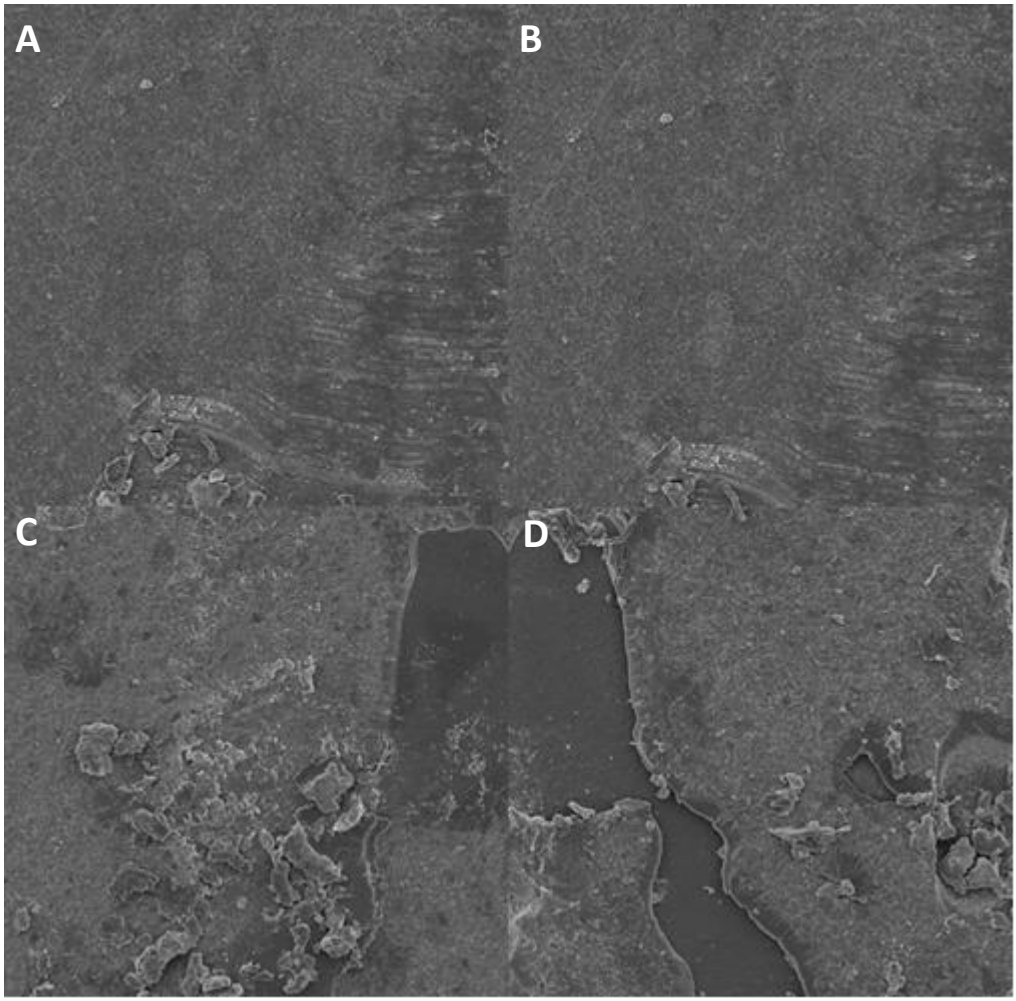

Fig. 7- Fracture pattern on both sides from EN group. Paired images (AB and CD) show the two debonded surfaces of the same stick. No distinction on the fracture patterns between all experimental groups was found. Images A and B show a uniform cohesive failure in resin cement with few areas with resin cement particles agglomerated; Images C and D show a mixed fracture pattern, with mainly fractured resin cement sites, many areas with resin cement particles agglomerated and some areas of adhesive debonding.

CONCLUSIONS

Within the limitations of this in vitro study the following conclusions can be drawn:

1. Different hydrofluoric acid etching concentration and time of application did not influence on the bonding effectiveness.
2. Self-etching ceramic primer effectiveness was comparable to hydrofluoric acid etching despite producing less surface morphological alterations.
3. Polymer infiltrated ceramic exhibit better bonding performance than lithium disilicate reinforced ceramic and leucite-based ceramic.

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3. DISCUSSÃO

Com base nos resultados deste estudo, a hipótese 1 (distintos protocolos de condicionamento induziriam diferenças na adesão dos cimentos resinosos com as cerâmicas vítreas) foi rejeitada, uma vez que a resistência da união não foi afetadas pelos tratamentos de superfície. Entretanto, a hipótese 2 (o tipo de material testado induziria diferenças na resistência da união e na morfologia de superfície) foi confirmada.

Os protocolos de condicionamento avaliados, neste estudo, não mostraram diferenças estatísticas entre si em relação à resistência da união (Tukey p> 0,05), embora maiores concentrações e tempos de aplicação de ácido fluorídrico (HF) possam aumentar a rugosidade (Tian *et al*, 2014) e a molhabilidade (Venturini *et al*, 2015), das superfícies dos materiais. Com base nisso, pode-se inferir que provavelmente não há correlação positiva direta entre a rugosidade da superfície e a resistência da união. Assim sendo, o aumento da exposição da cerâmica ao HF é desnecessário.

Uma possível explicação para isso tem relação com a maior profundidade de dissolução da fase vítrea promovida pelo aumento do tempo de condicionamento ácido associada a uma penetração deficiente do cimento resinoso devido à sua alta viscosidade (Naves *et al*, 2010). Além disso, a literatura sugere que a adesão química é a principal responsável por manter a união entre a cerâmica e o cimento resinoso na presença de aplicação de forças (Murillo-Goméz FJ, 2017).

O mecanismo de condicionamento do ácido fluorídrico não se baseia na corrosão da matriz vítrea da cerâmica, visto que o HF é um ácido fraco em termos de pH. No entanto, a fundamentação da decomposição vítrea da superfície cerâmica está na interação química entre os íons de fluoreto do HF e íons de silício da cerâmica (Tian *et al*, 2014). Isto sugere que a reação é íon-dependente e a exposição exagerada da superfície cerâmica ao HF não traria benefícios adicionais.

Os efeitos do HF sobre as propriedades mecânicas das cerâmicas são controversos. Embora alguns estudos não demonstrem alterações significativas na resistência à flexão de cerâmicas condicionadas (Fraga *et al*, 2015; Posritong *et al*, 2013), outros estudos mostraram uma redução significativa desta propriedade mecânica em diversos tipos de cerâmicas, mesmo em tempos menores de aplicação do HF (Zogheib *et al*, 2011; Addison *et al*, 2007; Hooshmand *et al*, 2008). Para evitar

os riscos de comprometer a resistência mecânica devem ser encorajadas formas menos agressivas de condicionamento da superficie das cerâmicas.

Neste estudo, foi observado que o padrão de condicionamento produzido pelo primer cerâmico autocondicionante (MBEP) foi menos pronunciado em relação àquele produzido pelo HF (Figuras 2;3;4), ao mesmo tempo que se mostrou ser eficiente na promoção da adesão. Este primer apresenta uma seqüência de aplicação simples e rápida devido ao menor número de etapas e não apresenta riscos de toxicidade do HF (Volkel e Braziulis, 2015). Embora existam poucos estudos na literatura sobre a eficácia do MBEP, este estudo mostrou resultados promissores até o momento. Não houveram diferenças estatísticas na μTBS do MBEP quando comparado aos outros tratamentos de superfície avaliados, em todos os materiais. Isto confirmou que a leve alteração da superfície cerâmica produzida pelo MBEP aliada a união química é suficiente para resultar em uma adesão eficiente (Murillo-Goméz FJ, 2017). Portanto, nossos resultados estão de acordo com os dados fornecidos pelo fabricante (Volkel e Braziulis, 2015), que afirmaram que este produto não só propicia benefícios adicionais em geral quando comparado com HF, mas que também mostrou-se eficiente na promoção de resistência da união. Estudos a longo prazo devem ser realizados para comprovar a manutenção da sua eficiência.

Em relação aos materiais utilizados neste estudo, EN mostrou os valores superiores estatisticamente de μTBS seguidos por DL e LC que não diferiram entre si (Tabela 2). Uma possível explicação para o maior valor de μTBS observado para EN quando comparado ao DL e LC está na superfície mais rugosa favorecida pela composição do material (Lise *et al*, 2017; Flury *et al*, 2017), na qual poderia mecanicamente auxiliar na união, especialmente nos grupos controle. A dissolução da fase vítrea de EN expõe a matriz polimérica, revelando uma estrutura em forma de “favo de mel” (Figura 4). Esta configuração estrutural pode oferecer um maior potencial para retenção micromecânica (Mainjot *et al*., 2016), bem como a possibilidade de radicais livres da matriz polimérica se unirem aos grupos metacrilatos do cimento resinoso, embora isso ainda não seja confirmado. Outra explicação possível pode ser encontrada nas propriedades mecânicas dos materiais. Enquanto DL e LC têm maior módulo de elasticidade, EN quando alongado é resistente a altos níveis tensão antes da falha, devido à sua rede integrada de polímeros (Swain MV, 2013). Isso pode significar que as tensões geradas na

interface sejam melhor distribuídas ao longo do corpo do material durante o teste de tração (Elsaka SE, 2014).

Da mesma forma que os resultados de resistência da união, a prevalência do padrão de fratura observado não diferiu entre os tratamentos de superfície avaliados. O modo de fratura predominante foi misto, seguido de coesiva no cimento, sendo EN o único material que apresentou falha coesiva em cerâmica (Figura 1). Nos grupos controle de DL e LC, todos os espécimes apresentaram desunião da interface antes do teste de μTBS, o que indica ausência de adesão entre os materiais nesses grupos. Surpreendentemente, o modo de falha mais prevalente para o grupo de controle de EN não foi adesivo, o que enfatiza as características inerentes à composição deste material.

4. CONCLUSÃO

Com base nos resultados encontrados neste estudo, pode ser concluído que:

1. Diferentes concentrações e tempos de aplicação de ácido fluorídrico não influenciaram na eficiência da adesão.

2. A resistência da união gerada pela ação do primer cerâmico autocondicionante foi semelhante àquela produzida pelo condicionamento com ácido fluorídrico e silano, apesar de produzir menos alterações morfológicas na superfície.

3. A cerâmica infiltrada por polímero exibiu uma melhor performance na resistência da união do que as cerâmicas à base de dissilicato de lítio e leucita.

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1 De acordo com as normas da UNICAMP/FOP, baseadas na padronização do International Committee of Medical Journal Editors - Vancouver Group. Abreviatura dos periódicos em conformidade com o Medline

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